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TEXTURAL AND STRUCTURAL REGULARITIES OF DEFORMATION BY ROLLING OF Zr-2.5%Nb ALLOY

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A review of studies of regularities of changes in the crystallographic texture of the longitudinal and cross rolling of the Zr-2.5%Nb alloy is given. Accordingly with this, plates were made from longitudinal fragments and rings of a tube ∅15.0×1.5 mm from the fuel assemblies of Russian RBMK reactors. The plates were annealed at 580 °C for 3 h. X-ray studies of texture changes with the degree of deformation of the plates by rolling were carried out. The principle of method of inverse pole figures was used with the calculation of the Kearns texture coefficients, as well as measurements of tilt curves (0002) was. Non-uniformiity of deformation during straightening of the tube blanks was taken into account. It is found that the main rotations of crystallographic *c*-axes in the plates at any rolling direction are carried out in the plane corresponding to the cross-section of the original tube. Existence of an initial stage of increased textural changes during deformation of the plates is found, what is qualitatively and quantitatively related to activity of tensile twins of the $\{10\bar{1}2\}[\bar{1}011]$ system. Regularities in texture changes at the subsequent moderate stage of deformation with the participation of compression twins are considered. The purely twinning nature of the textural doublet, which latter is characteristic of the rolling texture of metals of the titanium subgroup, is explained. It is noted and explained a fact that during cross rolling of plates (made from tube rings), textural changes occur at a rate twice as high as during longitudinal rolling. As considered, all similar regularities can be carried out for the metals of the titanium subgroup.

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INTRODUCTION

The titanium subgroup metals and their alloys with a hcp lattice are widely used in nuclear technology, aviation, medicine, and other industries. On their basis, making of semifinished products and products with desired properties by deformation requires certain knowledge concerning also textural regularities. In developing the theory of deformation of such metals, it is important to know, in particular, what role does twinning play in the development of texture and, in general, in their deformation. In this, the distinctive feature of this group of metals – hcp structure of their lattice and certain ratio of the main crystallographic axes (c/a = 1.58…1.59) – is significant.

Since the mentioned structural features of the hcp metals of the titanium subgroup practically coincide, there should be a certain commonality in the regularities of development of the crystallographic texture of deformation of such metals in their pure state. This also must refer to high-purity titanium. The aim of this work is to reveal the textural and structural regularities of cold deformation by rolling such metals in the example of Zr-2.5%Nb alloy, what is a structural material for thermal nuclear reactors.

EXPERIMENTAL

In the ideal case, original samples for such studies should be plates with a uniform fine-grained structure. However, to obtain them from an ingot, significant deformation is required. In this case, a high stationary texture is established in them and the initial regularities of texture formation are lost.

A more moderate texture appears in annealed tubes made of such metals, where its quantitative index – the Kearns coefficient [1] – is almost equally distributed between the radial and tangential directions of the tubes. In this work, the material of the tube ∅15.0×1.5 mm from Zr-2.5%Nb alloy, used as one of the elements of fuel assemblies of Russian RBMK reactors, was studied. Preliminary texture studies had shown that such a tube (after standard annealing at 580 °C for 3 h) has a relatively more moderate texture with a fan-shaped angular distribution of crystallographic *c*-axes (the main texture attribute) within deviations of ~ 30º from the tube cross-sectional plane. Longitudinal fragments and rings 10…12 mm wide were cut out from such a tube and then were straightened. The made original plates were annealed at 580 °C for 3 h or more in a vacuum of 10^{-5} mm Hg ($1.5\cdot10^{-4}$ Pa) and then their surfaces were etched [2]. The plates had ~1.35 μm initial thickness.

Then the plates were rolled at room temperature from 6 to 55% with a step of 5...7% and at a speed of 5...10 s^{-1} without intermediate annealing. According to the characteristic features of texture of the original tube [2], the rolling was considered as an analogy of longitudinal rolling for plates made from the longitudinal fragments, and as cross rolling for the plates from the rings.

Texture studies were carried out on a DRON4-07 X-ray diffractometer in CuK_α radiation with the proportional counter. Texture analysis was performed using the method of inverse pole figures (IPFs [3]) with calculation of pole densities (P_{ji}) from the intensities (I_{ji}) of *i*-th reflection lines at measurement on *j*-th direction:

$$P_{ji} = (1/R)\sum_i I_{ji}/I_{0i} \quad \left[R = \sum_i A_i I_{ji}/I_{0i} \right], \qquad (1)$$

wherein I_{0i} are the calculated or experimental values of the intensities of the material in the non-textured state; A_i are the statistical coefficients of the pole distribution over orientations in the crystal lattice [4].

P_{ji} are analogous to the density of orientations (*hkil*) in *j*-th measurement direction. Based on a $\{P_{ji}\}$ group, the Kearns texture coefficient *f* [1] was calculated for a given measurement direction:

$$f_j = \sum_i A_i P_{ji} \cos\alpha_{ji}, \qquad (2)$$

wherein α_{ji} is the angle between the measurement direction (normal to the measurement plane) and the orientations of crystallographic *c*-axes of the reflecting group of grains.

The Kearns coefficient is an integral indicator of the directionality of *c*-axes relative to the measurement direction under consideration. In the main, this is the normal to the plane of the plates, i.e. the normal direction (ND).

A primary stage of research is to determine regularities of change in the Kearns coefficient with degree of deformation. During the research, it was found that the graphs of such changes for different sides of the plates are different. This was associated with an effect of non-uniformity of the straightening deformation of tube blanks that is expressed with mutually opposite values of near-surface deformations in the plates. This effect takes place because of low penetrating depth of X-ray radiation in the material (~ 10 μm). Let us agree to call these surfaces either "front" or "back" corresponding to the outer and inner sides of the original tube. The degree of initial surface deformation in the plates was estimated from the geometrical parameters of the tube. Practice has shown sufficient accuracy of the result of introducing such corrections to the cross rolling of plates – as a "plus or minus" term ±(10±0.5)%, – since in its direction the deformation of their straightening, including also its different signs, coincides with the subsequent deformation.

An analysis of pole density distributions of the plates relative to ND and, in particular, recording of tilt curves was also carried out.

RESULTS AND DISCUSSION

Fig. 1 shows graphs of changes in the Kearns coefficient of alloy plates with the degree of deformation by their longitudinal and cross rolling (as this denoted above in relation to direction of rolling which had carried out on the original tube) [2].

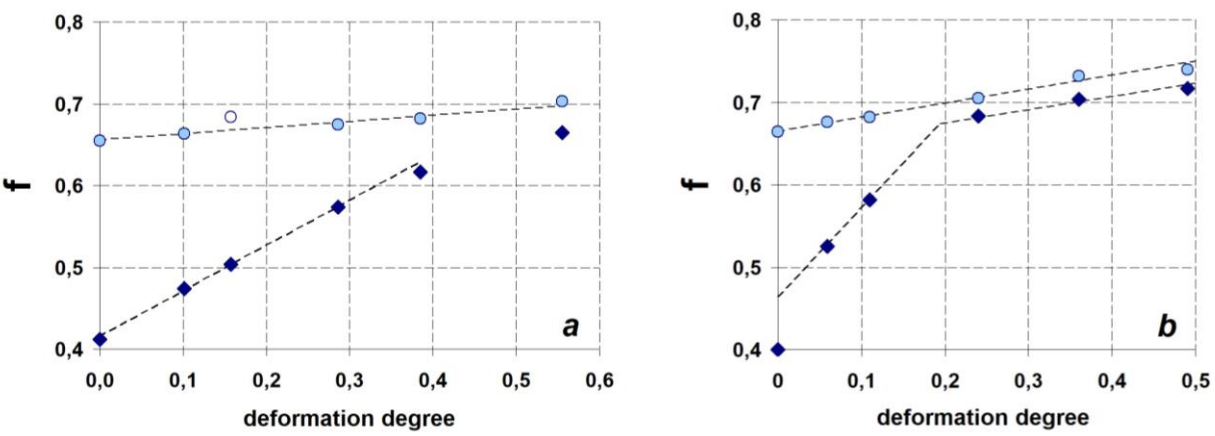

Fig. 1. Dependences of the Kearns coefficient on deformation by (a) longitudinal and (b) cross rolling on the front (●) and back (◆) sides of the plates [2]

In addition to the differences between Fig. 1, their graphs within each figure, as noted above, differ for different sides of the plates. Taking into account the sign, adding the corresponding corrections to the cross rolling will satisfactorily match graphs 1b, because in this case, the directionality of the deformations coincides. The initial point falls out of the regularity (see Fig. 1,b). This is associated with the residual viscoelastic effect after straightening of the plates, in particular, with the fact that the annealing temperature was not high enough to eliminate it. This effect manifested itself at the initial act of deformation.

It should be noted that Fig. 1 are characterized by an almost twofold difference in the corresponding graphs in terms of deformation scales. In particular, the correction for the non-uniformity of initial deformation of longitudinally rolled plates (see Fig. 1,a) should also be doubled. In fact, it is ±0.25...0.30%.

If to present the graphs of changes in the Kearns coefficient with resulting deformation, i.e. the deformation of their near-surface layers, taking into account corrections, then a clear two-stage nature of such graphs can be noted as a characteristic feature of uniform textural changes in the plates. So, for the initial stage of the graphs, a significant rate of increase in the Kearns coefficient is inherent. In this case, the deformation limit of existence of the initial stage for non-textured original material, according to estimates, should not exceed 50...60%. In particular, for plates deformed from ingots, the initial stage of changes would be practically absent, as mentioned above.

To present further analysis, tilt curves for reflections (0002) relative to ND along the plane of the longitudinal (N–R) and transverse section (N–T) of the plates at the both directions of rolling are shown in Fig 2 [5]. The tilt curves were taken from the back sides of the plates and thus represent the second, stationary stage of texture changes (see Fig. 1). The curves represent the distribution of *c*-axes in the coordinates of deviation angles (α) from ND normal along the corresponding sections, and displayed with vertical intervals.

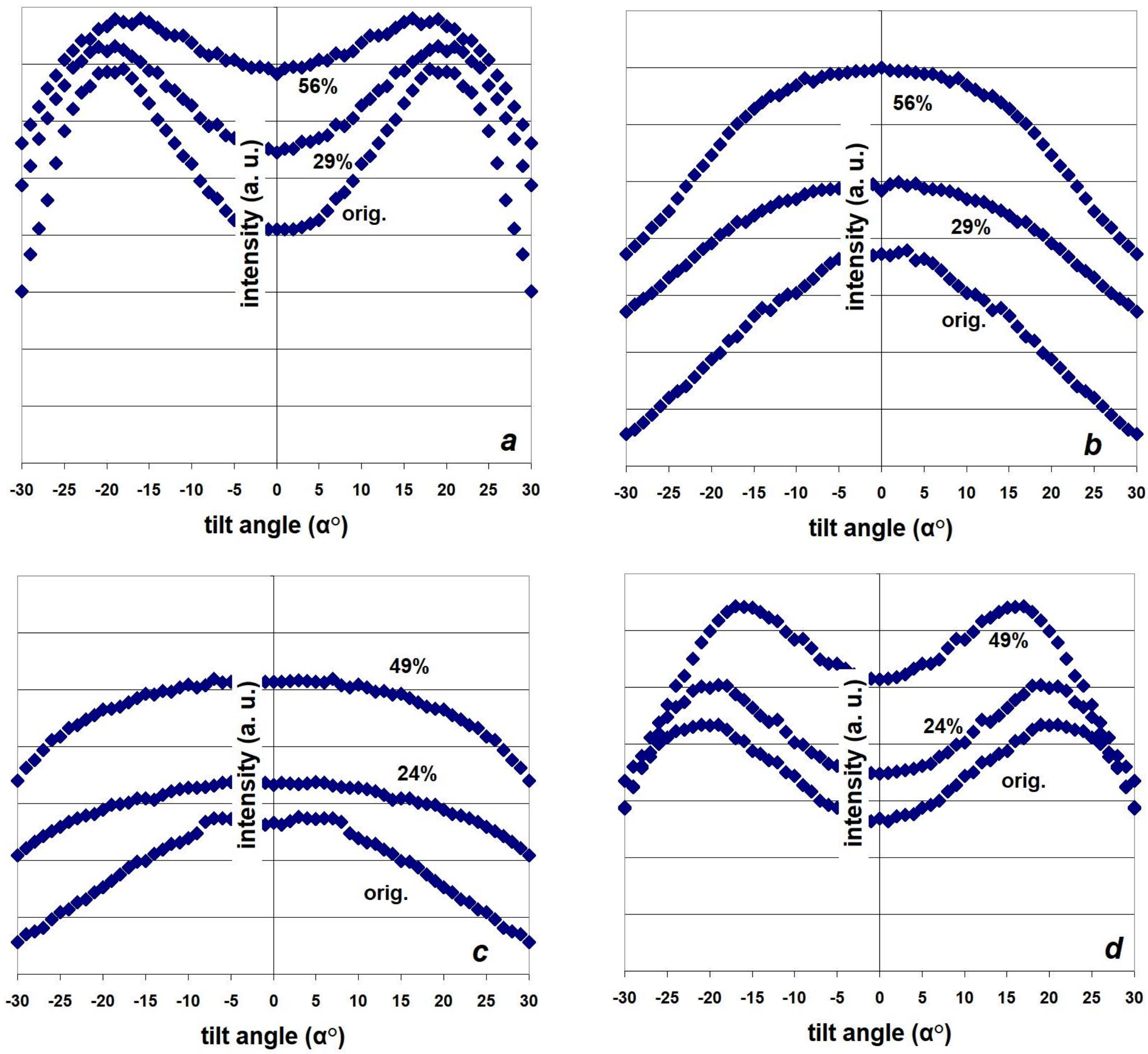

Fig. 2. Distributions of c-axes orientations in the planes along the longitudinal (a, c) and cross section (b, d) of the alloy plates after longitudinal (a, b) and after cross rolling (c, d) [5]

For longitudinal rolling, the curves represent a typical rolling texture, with the usually specific texture doublet in cross-section. A distinctive feature of cross rolling can be briefly described as switching the directions associated with texture formation: (N–T) ↔ (N–R). In other words, in the studied area of deformations, the texture and its changes are mainly concentrated within deviations of *c*-axes by ±30º from the cross-sectional plane of the original tube.

Further, the results of study of changes in the coefficients f_j (2) of these plates in two other directions, mutually orthogonal to the ND, are presented: RD is the rolling direction, TD is the transverse direction. The results are shown in a ternary diagram (Fig. 3). The value of f_{ND} has averaged, being determined by the formula $f_{ND} = 1 – f_{RD} – f_{TD}$ for each of the plates.

The nature of the changes in f_j (see Fig. 3) confirms the previous conclusions about the rotations of the *c*-axes of the plate material along the cross-section of the original tube. So, when “switching” the directions RD ↔ TD for any graphs, their courses will almost coincide.

The manifestation of “switching” of this kind (see Figs. 2, 3) is not sufficiently understood. It remains to be assumed that such directionality reflects the specifics of the annealed state of the tube material and even its straightened fragments. To clarify, a temperature of 580 °C is not enough to completely remove the residual viscoelastic stresses in the material, that stimulate such directionality of texture changes.

The structural nature of the textural changes is also not clear enough. However, taking such commutation as a fact, one can explain the difference in the rates of these changes between the two geometries of rolling. So, in each of both cases, the general dynamics of *c*-axis turns is closely related to the specifics of the deformation along the corresponding planes of turns. So, since for cross rolling this is the ND–RD plane, and the actual deformation Δ in this plane is accompanied by a combination of compression along ND and tension along RD, then for longitudinal rolling this is the ND–TD plane, and the actual deformation in this plane is accompanied only by compression along ND with the corresponding deformation analogue Δ/2. This explains the twofold difference between Fig. 1 in all length of the graphs along the deformation axis.

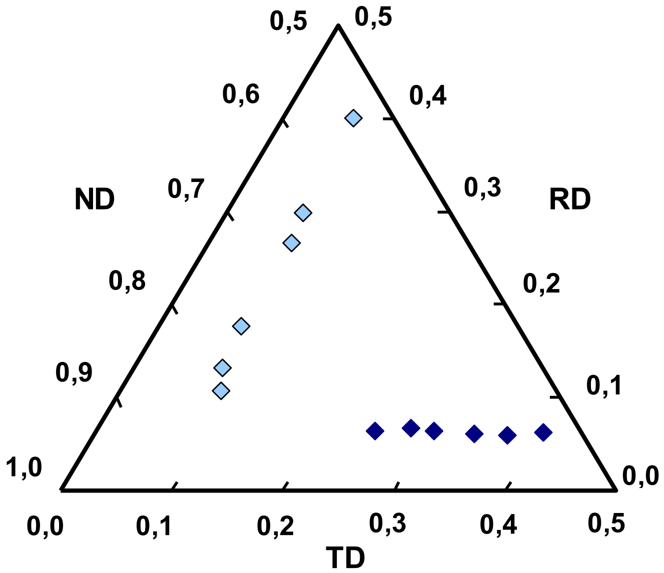

Fig. 3. Ternary diagram of changes in texture coefficients f_j in basal tripod of directions associated with the longitudinal (◆) and cross rolling (◆) of the investigated stacks of Zr-2.5%Nb alloy plates

To clarify the structural nature of the initial stage of the graphs (see Fig. 1), the volume fraction of the aggregate of grains with the *c*-axes deviations within ±30º from the plane of the original and deformed plates was calculated: $c_p = \Sigma A'_i P_i$, where A'_i is an analogue of the solid-angular space of orientations of the axes of the *i*-th reflecting grains falling into the given area (±30º). Fig. 4 shows the relationship between the Kearns coefficients and the corresponding c_p values. The linearity of the graphs in Fig. 4 and their slope that almost surely is -45º indicates that all the portions of orientations of *c*-axes directly, without intermediate positions, consequently go, simply speaking, from the plane of the plates to the position of the normal to the surface of the plates (ND). In other words, the *c*-axes are reoriented abruptly through an angle near 90º.

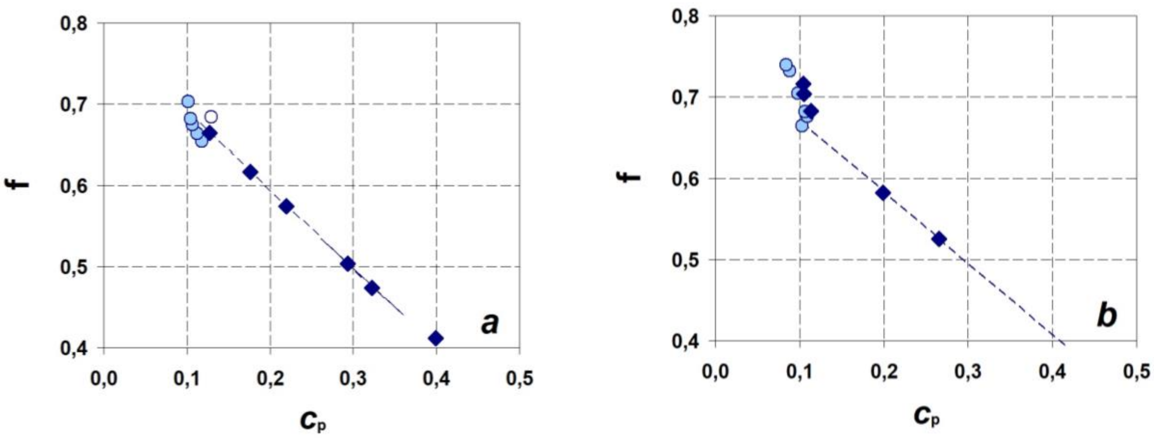

Fig. 4. Correlation of the Kearns coefficients (see Fig. 1) with the proportion of c-axes orientations near the plate plane directions – for the longitudinal (a) and cross rolling (b) [2]

As is known [6], such turns can be carried out only by the $\{10\bar{1}2\}[\bar{1}011]$ system of tensile twins. This is shown in Fig. 5 by illustration of the schemes of by-twin rotation of hcp lattice by 85.2º (see or 94.8º; Fig. 5,a) and of the geometry of the grain twinning (see Fig. 5,b).

Since such turns are carried out in the plane of the cross section of the original tube, then, as a consequence, Fig. 5 is most of all related to cross rolling. At the same time, Fig. 5,b illustrates the changes with the degree of deformation Δ by twinning of an ideally oriented grain: the proportion of the twinned part of the grain is $-\Delta c_p = \Delta' = \Delta/2\sin^2(94.8°/2) = 0.92\Delta$. As a result, for the grain, $\Delta f = -\Delta c_p \cdot \sin 85.2° = -\Delta c_p = 0.92\Delta$. With such results, the slopes of the graphs in Fig. 4 and the graphs of the initial stage in Fig. 1,b (and also 1,a) are practically agreed, since in this case – for an arbitrary ensemble of grains – all operated quantities – Δf, Δc_p and Δ – are multiplied by initial concentration of grains will be twinned.

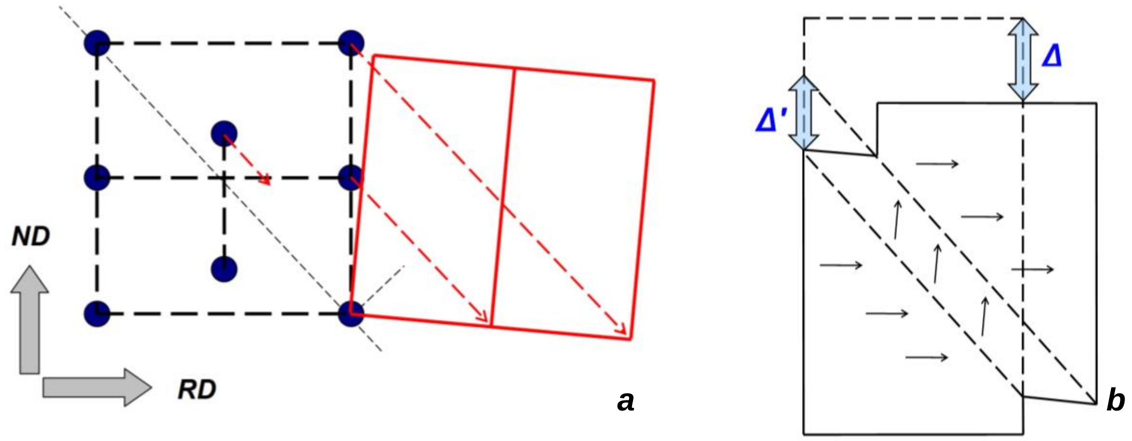

Fig. 5. Twinning by the $\{10\bar{1}2\}[\bar{1}011]$ *system during grain deformation in zirconium and its alloys*

Thus, the first stage of texture changes, which is absent in plates made from bulk blanks by the traditional mechanical method, is almost completely associated with the activity of tensile twins of the $\{10\bar{1}2\}[\bar{1}011]$ system.

To analyze the structural processes at the second stage of texture changes, the dynamics of angular distributions of *c*-axes relative to the ND of plates is considered [5]. Such distributions were built for each degree of longitudinal and cross deformation of the plates concerning the P_i values for reflections $(h0\bar{h}l)$: $P_i \equiv P_{(h0\bar{h}l)} \equiv P(\alpha)$ (1), (2), and are displayed in Fig. 6 in the representation of $\ln P(\sin^2\alpha)$ with vertical intervals $\Delta \ln P = 3$.

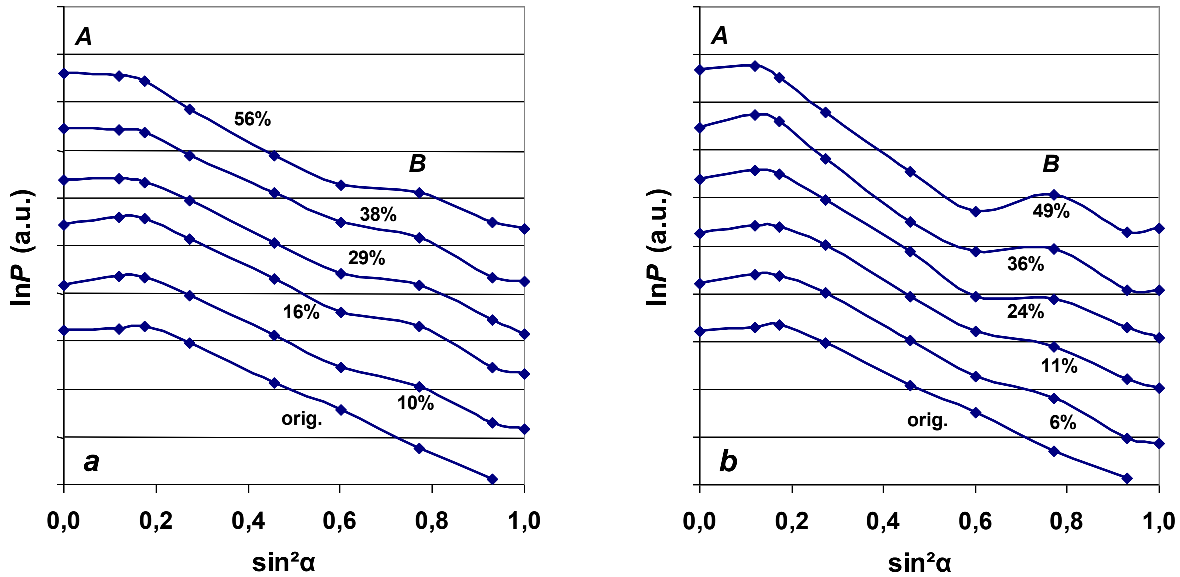

Fig. 6. Representation of P(α) distributions for the $(h0\bar{h}l)$*-planes group based on measurements on the back sides of the longitudinal (a) and cross rolling plates (b)*

According to Fig. 6, the basis of $\ln P(\sin^2\alpha)$ distributions is described by straight lines. In other words, the distributions themselves are described by a Gaussian of type $P(\alpha) = a\cdot\exp(-b\cdot\sin^2\alpha)$. On this basis, *A* and *B* points (see Fig. 6) represent some irregularities. The existence of such irregularities is confirmed by studies of pure zirconium and hafnium plates [7] obtained directly from ingots. It can be assumed that such irregularities are created by some fraction of discrete reorientations of *c*-axes from *A* ($\sin^2\alpha = 0$) to *B* position ($\sin^2\alpha = 0.7…0.8$; $\alpha = 55…65°$).

According to the data obtained and existing information [5, 6], such a reorientation in the plates of Zr-2.5%Nb alloy can be attributed to action of compression twins of the $\{10\bar{1}1\}[\bar{1}012]$ system ($\alpha = 57.1°$), what manifests itself within a series of reflections from crystallographic planes $(h0\bar{h}l)$ (see Fig. 6), including the $\{10\bar{1}1\}$ twinning plane. Activity of compression twins of the $\{11\bar{2}2\}[\bar{1}\bar{1}23]$ system ($\alpha = 64.2°$) is considered to be more preferable [8, 9]. However, it is impossible to fix this with methods presented here.

There is an assumption that the doublet (see Fig. 2) in usual rolling texture is formed by a twin translation of orientations from *B* point to the neighbourhood of *A* point [5]. More exactly, it does to the bending point of the graphs in Fig. 6: $\sin^2\alpha = 0.14…0.15$; $\alpha = 22…23°$. In Fig. 2, this is the position angle of the maxima – ±20…22°. Such a rotation could be carried out with activity of $\{10\bar{1}2\}[\bar{1}011]$ compression twins according to the scheme $85.2° – \alpha = 63…64°$.

Thus, the participation of compression twins of the second system fits into this scheme, and a series of turns from ND position is carried out according to the scheme $\{10\bar{1}2\}[\bar{1}011] \rightarrow \{11\bar{2}2\}[\bar{1}\bar{1}23]$. Such participation changes the plane of the previous reorientation by an angle of 30° and thereby takes the *c*-axes out of the main plane of texture changes, what can be seen in Fig. 2.

On the other hand, the texture doublet can simply be considered as the result of a violation of the linearity of the graphs in Fig. 6 at *A* points as a result of primary rotations $A \rightarrow B$. This does not exclude the previous pattern of rotations, especially since it fits into such linearity. For it actually falls on the border of the linear section, and this can be considered a special case of this. Generally speaking, the statistical nature of such linearity can be provided by an extended variety of turns, in particular, by the additional participation of twins of the $\{11\bar{2}1\}[\bar{1}\bar{1}26]$ system ($\Delta\alpha = 34.8°$ [5, 6]) and sliding mechanisms that can contribute to regular linearity of graphs.

In any case, the effect of formation of texture doublet during rolling of alloy plates may be associated exclusively with twinning processes.

The geometric conditionality of the revealed regularities gives reason to conclude that as a whole such regularities should manifest themselves in metals of the titanium subgroup. Using the example of niobium as an alloying element of this alloy, there is reason to believe that such metal additives or impurities of the order of a percentage will not violate the quantitative characteristics of textural changes. In particular, this is confirmed by several results of studies of the rolling texture of the Zr-1.0%Nb alloy.

At the same time, the presence of interstitial impurities in these metals, even less than half a percent, can significantly affect textural changes, at least increase the rate of textural changes at the initial stage up to two times [10]. This is a sign of additional activation of twinning due to impurity suppression of slip. For example, this can take place in grade BT1-0 (Rus.) technical titanium, which impurities are an order of magnitude higher than in reactor zirconium alloys.

SUMMARY

Results of X-ray studies of the crystallographic texture of the longitudinal and transverse rolling of Zr-2.5%Nb alloy plates are presented. A distinctive feature of the work was investigations in the area of moderate initial texture of the material. In this regard, the subject of research was plates obtained from fragments of ∅15.0×1.5 mm tube, which is one of the elements of fuel assemblies of Russian RBMK reactors. To analyze the results, the method of inverse pole figures was used with calculations of the Kearns texture coefficients. Corrections were calculated to take into account the deformation non-uniformity which caused by straightening of tube fragments had been.

Due to the moderate texture in plates with a deformation of up to 20...40%, the initial stage of changes in the Kearns coefficient for the plate rolling plane was revealed. It is the stage of an accelerated rate of its linear changes with degree of deformation in the near-surface layers of the plates.

For the both rolling geometries for the alloy plates, it has been shown that the texture changes by rotations of *c*-axes along the cross-section of the original tube. An explanation is given for this directionality as well as for the experimental fact that, for all the deformation degrees of alloy plates, the rate of texture changes during cross rolling is two times higher than during longitudinal rolling of them.

It was shown that the initial stage of growth of Kearns coefficient is entirely associated with primary tensile twinning by the $\{10\bar{1}2\}[\bar{1}011]$ system, with rotation of *c*-axes of hcp lattice of the alloy by 85º.

A numerical relationship was revealed between the textural changes in the alloy plates at the initial stage, degrees of rolling, and the twinning process.

Two concurring twinning models for formation of the texture doublet on the second stage of texture changes during rolling of the plates are proposed. These models include activity of compression twins – without noticeable participation of slip.

It is considered that in similar way all the regularities can be carried out for metals of the titanium subgroup.

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ТЕКСТУРНІ І СТРУКТУРНІ ЗАКОНОМІРНОСТІ ДЕФОРМАЦІЇ ПРОКАТКОЮ СПЛАВУ Zr-2,5%Nb

Д.Г. Малихін, В.М. Грицина, К.В. Ковтун

Проведено огляд закономірностей змін кристалографічної текстури поздовжньої та поперечної прокатки сплаву Zr-2,5%Nb. Згідно з цим було виготовлено та відпалено пластини з поздовжніх фрагментів та кілець труби ∅15,0×1,5 мм з паливної збірки реактора РБМК. Виконано рентгенівські дослідження текстурних змін зі ступенем деформації пластин. Застосовано прийоми методу зворотних полюсних фігур з розрахунком текстурного коефіцієнта Кернса, а також зйомки кривих хитання (0002). Враховано неоднорідність деформації при випрямленні трубних заготівок. Встановлено, що основні повороти кристалографічних осей ***c***, незважаючи на напрямок прокатки, відбуваються у площині поперечного перерізу вихідної труби. Встановлено існування початкової стадії підвищених текстурних змін з деформацією пластин, що кількісно узгоджується з активністю двійників розтягнення системи $\{10\bar{1}2\}[\bar{1}011]$. Закономірності текстурних змін на наступній помірній стадії віднесено до участі двійників тиснення. Обґрунтовано чисто двійникову природу текстурного дублета, який є характерним для прокатки металів титанової підгрупи. Встановлено та пояснено, що темпи текстурних змін при поперечній прокатці пластин (виготовлених з кілець) є у два рази більше, ніж при поздовжній. Вважається, що у цілому подібні закономірності повинні відбуватись у металах титанової підгрупи.